Intelligent Processing of High Performance Materials

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ABSTRACT

Intelligent Processing of Materials (IPM) is an emerging methodology for simulating and controlling the processing and manufacture of materials. It is finding widespread application during the manufacture of electronic, photonic and composite (i.e. high performance) materials, as well as primary metals such as steel and aluminum. IPM simulation tools seek to create, at the level of an engineering workstation, a “virtual” version of a process. They combine process models for a material’s response to processing stimuli (e.g. pressure, temperature, environmental conditions,...) with characteristics of the process equipment to predict the material’s performance defining attributes at the completion of the process. Thus, they provide a methodology for the process engineer to conduct “what if...” trials in minutes or hours compared to weeks or months of practical experimentation, and for near optimal processing approaches to be identified. IPM controllers are an innovative extension of today’s state-of-the-art in control technology. They exploit the recent availability of non-invasive sensors that sense critical product variables during the process. This new knowledge about the state of the process, together with process models, can be used to plan and execute feedback control schemes leading to products with “goal state” combinations of performance defining property attributes. Using the consolidation processing of Ti-24Al-11Nb and Ti-6Al-4V matrix composites reinforced with SCS-6 fibers as an example, it is shown that this radically new approach to (materials) process control enables the processing of high performance materials hitherto considered too unstable for commercialization and significantly improves the yield/quality of others already being manufactured today.
1. INTRODUCTION

The two aims of this paper are to describe an “idea” that has come to be called Intelligent Processing of Materials and to review ongoing efforts to apply it to the processing of high performance composites. IPM has recently emerged as a new technology for designing and controlling the synthesis and processing of materials\(^{(1)}\). It is having a pervasive impact upon the design and manufacture of many of the advanced materials under development today for military and civilian systems. For example, it is used during the growth of ceramic fibers for high performance composites\(^{(2)}\), for the production of novel alloy powders used in aircraft engines\(^{(3, 4, 5)}\), during the manufacture of metal and ceramic matrix composites\(^{(6, 7)}\), for the deposition of coatings for oxidation protection\(^{(8)}\), and in the production of high thermal conductivity substrates for the thermal management of multichip modules\(^{(9)}\). It is also being widely explored by the defense electronics community to improve, for example, the yield and performance of infrared imaging sensors\(^{(10)}\) and to speed the development of new device technology based upon quantum well structures\(^{(11)}\). The primary metals industry also continues to vigorously pursue its application through collaborative programs with the National Institute of Standards and Technology (NIST) and the Department of Energy\(^{(12, 13)}\). The processes to which it has been applied are likewise diverse and include continuous casting, extrusion, steel refining, Bridgman and Czochralski crystal growth, inert gas atomization, spray deposition, hot isostatic pressing, chemical vapor deposition, molecular beam epitaxy, etc.

2. IPM OVERVIEW

So what is IPM, and why is it attracting so much interest today? IPM can most easily be described with the aid of a sketch that schematically illustrates both the design and the control of one processing step during the manufacturing of a material, Figure 1. Let’s begin at the lower right of the diagram with the box “Goal-State Property Envelope”. Much of the motivation for IPM’s development has come from the growing realization that successful materials are carefully engineered products that possess an optimum balance of properties (including cost) that make them best suited to a particular type of application. For example, Nextel 610\(^{\text{TM}}\) alumina fibers recently developed by the 3M Company have extraordinarily high strengths (and stiffnesses) at low and intermediate temperature, because they have a fine (nanocrystalline) grain size and contain few defects. They are also chemically compatible with aluminum alloys, and are reasonably inexpensive. This particular combination of properties is almost ideal for directionally reinforcing aluminum alloys\(^{(14)}\). However, the fine (nanocrystalline) grain size that imparts low temperature
strength to Nextel fibers results in significant creep at high temperatures (above say 1000°C) and other more creep resistant fibers would be necessary to ensure dimensional stability of other types of metal matrix composites (MMC) intended for higher operating temperatures. Furthermore, if the fiber’s cost is too high, designers may choose to select a conventional high strength alloy steel, nickel, or titanium alloy instead of the aluminum matrix composite and trade off the penalty in performance against system cost. In fact, all materials in service today face intense competition of this type and markets only exist for those that satisfy the increasingly stringent needs of designers whose products must now compete in a global marketplace.

**INTELLIGENT PROCESSING:**
Interactive guidance of material microstructure (properties) during processing toward a known goal state

![Diagram](image)

**Figure 1.** The IPM loop for a hypothetical materials processing step.

Turning again to Figure 1, we see that a balance of properties leads to a “Goal-State Microstructure” because properties are, for the most part, composition and structure sensitive. Thus, the cut-off wavelength of a Hg$_{1-x}$Cd$_x$Te infrared detector depends critically on the value of $x$\(^{(15)}\), the oxidation rate of Si$_3$N$_4$ coatings varies by orders of magnitude when the grain size is changed\(^{(6)}\), and the damage tolerance of metal/ceramic matrix composites is lost when the fiber-matrix interface is allowed to form reaction
products\(^{16}\). An important issue here is that frequently the many properties of interest to a designer are each affected differently by composition/microstructure, and a delicate balance must be struck to obtain the needed property combinations. So we see that today’s successful materials must have an “engineered” composition/microstructure which meets a design’s specification. They should do this within the smallest possible tolerances so that conservative “design factors” can be eliminated or reduced, and the performance of the material fully exploited.

Now let us look at how the processes for materials synthesis and processing have been designed and controlled in the past. The box marked “Processing Environment” in Figure 1 symbolically represents any materials manufacturing process, e.g. it might be a microwave reactor used to deposit diamond wafers for thermal management applications\(^{17}\). Conventionally, a process like this would be controlled by sensing and attempting to hold constant, the variables of the process, i.e. reactant gas flow rates, microwave power, and the temperature of the substrate upon which deposition occurs. The set-points for these would have been established by trial and error and would correspond to those that gave the highest yield (and lowest cost) material that satisfied the design needs of let’s say a heat sink in a multichip module. When the windows in process space are wide, and the tolerances on microstructures large, this trial and error approach may lead expeditiously to an acceptable set of process conditions. In fact, most of the materials in use today have used this strategy quite successfully. But, when society tightens the microstructure tolerances and competition drives the development of new materials and processes with reduced “processibility windows”, the conventional approach begins to break down. This might first be recognized by a very low yield for the process. It is also characterized by long lead times to commercialization, questionable quality and very high cost. When placed in the context of today, where the market for materials is rapidly fragmenting into a multitude of small volume “niche” materials, we see that better approaches to processing are needed if we are to continue to field new materials at the rate of the recent past.

Intelligent Processing of Materials is one approach that is widely used today to win a high stakes game to become a part of tomorrow’s materials vendor base\(^{1, 18, 19}\). As a feedback control methodology, it is predicated upon the development of “microstructure sensors”, Figure 1. These are new sensor technologies originally developed by the nondestructive evaluation community, with a capability to directly sense the product being formed and allow inference of its quality defining attributes\(^{20}\). A good example is eddy current (proximity) sensors which can be used during the hot isostatic pressing of alloy powders\(^{21}\) and metal matrix composites\(^{22}\) to measure the remaining
porosity during consolidation. Other sensors have been developed for measuring liquid-solid interfaces during solidification of metals/alloys\(^{(23)}\) or semiconductors \(^{(24, 25)}\), texture in rolled steel/aluminum alloys\(^{(26, 27)}\), grain size during recrystallization\(^{(28)}\), fiber diameter\(^{(29)}\)/crystallographic orientation\(^{(30)}\) during single crystal sapphire growth, ... When the appropriate sensor technology exists, or can be developed at a reasonable cost, it becomes possible to perform feedback control on the product's microstructure and to directly attack the problem of variability.

However, this is is not as simple as implementing feedback control of process variables. For one thing, there is no direct way to control microstructure - the only variables available for direct actuation have to do with the processing equipment, i.e. heat flux distributions, gas/liquid flow rates, pressures, and so on. One way to use the new sensor measurements for feedback control is to know beforehand what is referred to as “Material Dynamics” in Figure 1, and use this for model-based control. The material dynamics are just a mathematical description (usually greatly simplified) of the process that predict the relationships between the variables one can directly actuate and the microstructural quantities of interest. This process modeling has been an area of intense study during the past two decades and excellent models for many of the phenomena encountered in processing now exist. For example, the density of alloy power preforms during hot isostatic processing (HIPing) or sintering can be predicted with models developed by Ashby and his coworkers\(^{(31)}\). The software for doing this on a personal computer is widely available\(^{(32)}\) and software products are being introduced for handling more complex near net shape problems\(^{(33)}\). By numerically integrating these models, one can even simulate the evolution of the process and so predict the material dynamics. Knowledge of the machine’s dynamics (i.e. its heating and pressurization rate limits in the HIP case) then allows the prediction of the outcome of any realizable process, and the possibility of implementing the two IPM modalities: designing optimal process schedules via process simulation and using model-based feedback control\(^{(34)}\) to achieve goal-state microstructures.

In cases where the models are difficult to develop or are too complex for treatment by the more formal methods alluded to above, other more heuristic approaches are being developed using neural nets\(^{(35)}\), fuzzy-logic\(^{(36)}\) and expert systems\(^{(37-39)}\). Indeed, it was an early reliance upon these artificial intelligence techniques that led to the coining of the term Intelligent Processing of Materials. It was not that the community engaged in its development were proposing that other methods were in some way unintelligent!
3. MMC PROCESSING

The designs of future high speed aircraft and propulsion systems have generated a need for stiff, strong, light, oxidation, and creep-resistant materials that can be used at high temperatures\(^{40, 41}\). These needs cannot be satisfied by the materials available today, and researchers are developing a variety of advanced composite materials composed of light metal/intermetallic matrices reinforced with silicon carbide or aluminum oxide fibers. The matrices of interest include conventional titanium (e.g. Ti-6Al-4V) or nickel alloys, and intermetallics based on Ti\(_3\)Al\((\alpha_2\) phase), TiAl \((\gamma\) phase), Ni\(_3\)Al, NiAl,....

Metal and intermetallic matrix composites are difficult to process. Contact between molten titanium alloys and the ceramic fibers for more than a second or two results in aggressive chemical reactions, fiber dissolution, and a loss of critical matrix/fiber properties\(^{42-45}\), which results in a serious reduction of composite strength\(^{46}\). A variety of novel processing approaches are being developed to bypass this. They include foil-fiber-foil\(^{47}\), vapor phase fiber coating\(^{48-50}\), powder cloth\(^{51}\), and molten spray deposition\(^{19}\). Each reduces or even eliminates liquid-fiber contact, but all require additional lay-up and consolidation processing to achieve a near net shape composite.

The use of molten droplet spray deposition methods has shown significant early promise for the deposition of conventional and intermetallic titanium alloys on prealigned ceramic fiber arrays\(^{19}\). The result is typically a 200\(\mu\)m thick monotape containing parallel, evenly spaced 140\(\mu\)m SCS-6 silicon carbide fibers in a porous matrix. The monotapes are layed-up to create a preform with the desired fiber architecture and then either hot isostatically or vacuum hot pressed to near net shape\(^{52, 53}\), Figure 2. The mechanical properties of the composites formed in this way are determined by the properties of the constituent materials, i.e. the matrix, fiber and interface and by residual stresses/damage induced during processing\(^{54, 55}\). In particular, the matrix may be depleted of desirable phases near the fibers\(^{56, 57}\), extensive chemical reaction of the fiber's coating can occur\(^ {58}\) and the consolidation process can cause bending or even fracture of the composite's fibers which has been predicted to result in a dramatic loss of strength\(^ {59}\).

The dependence of these phenomena upon processing have been analyzed and the material dynamics captured in process models and simulation tools. These have been used to explore the processibility of different composite systems, to identify optimum process schedules and, in conjunction with microstructure sensors, used for model-based feedback control. Each is now reviewed in more detail as an example of one way in which the IPM approach
can be usefully used, and to help identify where further efforts might be focused.

![Diagram of Single Asperity (GRVE) and Concentric Spheres RVE]

Figure 2. The complex deformation behavior of a monotape lay-up (left) can be treated by considering the micromechanical response of representative volume elements to simplified states of stress\(^{(53)}\).

4. CONSOLIDATION PROCESS SIMULATION

During the consolidation of a MMC monotape layup like that shown in Figure 2, densification occurs by the redistribution of matrix material. The fundamental mechanisms by which this is accomplished depend upon the temperature, applied load, and the matrix material. At low homologous temperatures and high pressures (relative to the material's yield stress), plasticity dominates; at high temperatures and low pressures, diffusional flow may be the most important mechanism while intermediate conditions activate power-law creep\(^{(53)}\). The local forces that drive material transport by these mechanisms within the layup are also able to bend and sometimes fracture the fibers\(^{(57)}\). This is mitigated by the use of high process temperatures which softens the material and facilitates consolidation with smaller local loads. However, prolonged high temperature exposure during processing results in chemical reactions between the fiber coatings and the matrix with undesirable consequences for both. It also has an adverse effect upon the frictional sliding coefficient of the interface\(^{(58)}\). All of these phenomena have complicated
(nonlinear) dependencies upon the process's actuatble variables (temperature, pressure) and are time and material system dependent. They are also influenced by the way in which the previous monotape deposition step was conducted (since this controls the porosity inside the tape and the roughness of its surface). It is a formidable assignment to experimentally establish all this, and this would have to be redone each time the composite system were changed (e.g. a fiber's strength improved, or a matrix alloy composition refined) or the conditions of the spray process varied. Because of this, the consolidation cycles used so far have been suboptimal resulting in composites of sometimes questionable quality\(^{54}\).

Recently developed process simulation software promises to overcome this\(^ {60}\). The simulations solve the fundamental micromechanical/chemical problems encountered in consolidation so that the material states can be calculated. The evolution of these states for any prescribed process cycle results in a temporal history of a composite's density, fiber fracture and fiber-matrix reaction.

### 4.1 Densification

Prior to consolidation, a plasma spray-deposited MMC monotape layup contains typically 35 to 45% internal porosity. Most of this porosity arises as a consequence of the surface roughness, the remainder being in the form of isolated voids within individual monotapes, Figure 2. Upon application of an applied stress, the layup densifies by the inelastic flow of material into the internal voids and by the deformation of surface asperities at places where adjacent monotapes are pressed into contact. Recently, a model has been developed\(^ {53}\) which can be used to predict the density of an MMC laminate as a function of the applied stress, temperature and time, given only the constituent materials properties and the geometrical parameters of the monotape (i.e. surface roughness distribution).

The complex problem is broken down by visualizing each composite monotape as consisting of two layers, or laminae, Figure 2. One, referred to as the “r”-lamina (reinforced lamina) contains the array of fibers, has all smooth sides and typically contains about 10% internal porosity in the form of isolated voids (the exact amount depends on how the spray process was performed), the other, referred to as the “s”-lamina (surface lamina), contains only the surface asperities. Densification of the s-lamina is treated in two stages: at lower relative densities \(D^{s_0} \leq D^s \leq 0.85\), where \(D^{s_0}\) is the initial relative density of the s-lamina, densification occurs by the blunting of asperities. This is referred to as “Stage I” by analogy to the situation encountered in powder consolidation\(^ {31}\). At higher densities \(0.95 \leq D \leq 1\), where D is the matrix
relative density), referred to as “Stage II”, densification is more accurately described as the shrinkage of internal, closed voids. A smoothed interpolation can be used to describe the transition between stages.

The model describing the Stage I densification of the s-lamina is based on an analysis of the contact stress required to cause blunting of a single hemispherical asperity of radius, r. The results of this unit cell analysis are given in Table 1 in the form of force-density relations for the cases in which the dominant deformation mechanisms are either plastic yielding, power-law creep, or diffusional flow. The overall response of the s-lamina is obtained by combining the predicted behavior for a single asperity with a statistical model describing the variation in size (r) and height (h) of the asperities on a typical monolam. Two probability distribution functions, \( \Phi_r \) and \( \Phi_h \), are introduced, representing the probabilities of encountering asperities of a given radius and height, respectively. The applied stress necessary to achieve a given densification rate (represented by the rate of change of s-lamina thickness, \( \dot{z} \)) is given as the sum of the forces acting on all asperities currently in contact multiplied by the total number of asperities per square meter, \( \rho \):

\[
\Sigma(z, \dot{z}) = \rho \int_{0}^{z_{0m}} \Phi_r \Phi_h F_c(h, r, D, \dot{D}) dh d\dot{h}
\]

Expressions for the probability density functions \( \Phi_r \) and \( \Phi_h \), are given in ref. (53) along with numerical values for statistical parameters used in the simulations presented below.

<table>
<thead>
<tr>
<th>Mechanism</th>
<th>Constitutive Law</th>
</tr>
</thead>
<tbody>
<tr>
<td>Plastic Yielding</td>
<td>( F_c = 2\pi r \left( h - z_0 \frac{D_0}{D} \right) \beta \sigma_0 )</td>
</tr>
<tr>
<td>Power Law Creep</td>
<td>( F_c = \left{ \frac{z_0 \left( \frac{D_0}{D} \right) \dot{D}}{\alpha \left[ rh - z_0 D_0 \right]} \right} )</td>
</tr>
<tr>
<td>Diffusional Flow</td>
<td>( F_c = \frac{r^2}{12 \pi \beta \omega(D) a_c(D) kT (z_0 D_0 / D^2 \dot{D}) \frac{D}{D_{ave} + 2 \rho D_{ave}}} )</td>
</tr>
</tbody>
</table>

The densification of the r-lamina (and of the s-lamina during Stage II) occurs by the shrinkage of internal voids; these are assumed to be spherical, non-interacting and of uniform size (implying that only homogenous stress
distributions are considered). The components of the strain rate are given by the gradient of a potential function, \( \Phi^m \) where \( m \) represents the assumed mechanism of deformation [i.e. plastic yielding (p), power-law creep (c) or diffusional flow(d)]:

\[
\dot{E}_{ij} = \Delta \frac{\partial \Phi^m}{\partial \Sigma_{ij}}
\]  

(2)

Here, \( \Delta \) is a scalar multiplier which may depend on the loading path. Potential functions used to describe the density-dependent material response for the cases of either plastic yielding, power-law creep or diffusional flow are summarized in Table 2.

**Table 2. Potentials used for Stage II densification**

<table>
<thead>
<tr>
<th>Mechanism</th>
<th>Potential</th>
</tr>
</thead>
<tbody>
<tr>
<td>Plastic Yielding</td>
<td>( \Phi^p = (\frac{\Sigma_c}{\sigma_0}) + (-2q(1-D)\cosh(\frac{3\Sigma_m}{2\sigma_0})) - 1 = 0 )</td>
</tr>
<tr>
<td>Power Law Creep</td>
<td>( \Phi^c = \frac{B}{n+1}(a(D)^2\Sigma_c^2 + b(D)^2\Sigma_m^2) )</td>
</tr>
<tr>
<td>Diffusion</td>
<td>( \Phi^d = \frac{\Omega}{kTR^3}\left{\frac{5\pi}{6}(\delta D_b + \frac{2R}{\pi}RD_c)\Sigma_c^2 + 2(\sigma D_b + \frac{3}{4}R_D D_c)\Sigma_m^2\right} )</td>
</tr>
</tbody>
</table>

The total principal strain rate components within each lamina are obtained as the sum of the contributions from each of the mechanisms:

\[
\dot{E}_i = \dot{E}_{pi} + \dot{E}_{ci} + \dot{E}_{di}
\]  

(3)

For the case of constrained uniaxial compression, the densification rate is then obtained from \( \dot{D} = -D\dot{E}_{kk} \).

The relative densities, for the s and r-laminae \((D^s, D^r)\), at time, \( t \), are computed as:

\[
D^s(t) = D^s_0 + \Delta D^s_p + \int_0^t \sum_{j=1}^2 s_j(\dot{D}^s_c + \dot{D}^s_d) d\tau
\]  

(4)

\[
D^r(t) = D^r_0 + \Delta D^r_p + \int_0^t (\dot{D}^r_c + \dot{D}^r_d) d\tau
\]  

(5)

where \( D^s_0 \) and \( D^r_0 \) are the initial densities of the s and the r-laminae respectively. As perfect plasticity is not a time-dependent mechanism, the
increment of densification due to plasticity for each lamina is calculated ($\Delta D_p$ and $\Delta D^p$) for every time step and added to the integral of the densification rate calculated for power law creep and diffusional flow.

Having calculated the relative density for the s and r-layers, the rule of mixtures is used to calculate the composite relative density ($D$):

$$D = D^s \cdot v^s + D^r \cdot v^r$$

(6)

where $v^s$ and $v^r$ are the volume fractions of the s and the r layers respectively.

A convenient way to show the densification response for a material is through the use of HIP maps popularized by Ashby\textsuperscript{(31, 32)} for powder consolidation. Figures 3 and 4 show examples for the Ti-24Al-11Nb (at%) and Ti-6Al-4V (at%) systems. These figures conveniently display the dependence of density upon pressure, temperature, and time, assuming the temperature and pressure are instantaneously applied and held constant.

4.2 Fiber Fracture

A fiber fracture model based upon micromechanical analyses of representative unit cells can be used to predict the overall response of a lay-up by calculating a sum of all the local (unit cell) responses\textsuperscript{(57)}. The general form of the unit cell chosen for analysis consists of a single fiber undergoing three-point bending due to forces imposed by (three) contacting asperities, Figure 5. An important parameter that determines the response of a given unit cell is the length of the fiber segment in bending which is governed by the statistically distributed asperity spacing. The deflection of the ceramic fiber, which is regarded as deforming elastically under all conditions, is calculated using simple beam theory and is a strong function of asperity spacing. From this deflection, the stress within the fiber can be calculated and, for a fiber of known diameter, elastic modulus and strength distribution, the probability of its fracture determined.

While the unit cell response is relatively easy to describe (only a single ODE is required to be solved), prediction of the overall rate of fiber fracture is quite complicated because as densification occurs, and monotapes are pressed together, the number of asperities in contact along the length of any given fiber continually increases. As these new contacts are established, existing bend segments become sub-divided into smaller (stiffer) cells. Thus, the statistical distribution of bend segment lengths is density-dependent and in order to calculate the number of accumulated fractures at any time, the creation, deflection and elimination of bend cells must be tracked. Elzey and Wadley\textsuperscript{(57)} have described an approach for this and developed a stochastic (Monte Carlo)
model that has been implemented throughout an entire process cycle. Although complicated in theory, the model predictions have been obtained in practice quickly and simply using Mathematica\textsuperscript{TM} operating on a fast (486 processor) PC.

![Graphs showing relative density versus pressures for various processing times.](image)

**Figure 3.** Relative density versus pressures for various processing times (in hours). The bolder lines denote the locus of points where two mechanisms equally contribute to the density\textsuperscript{53}.

![Graphs showing relative density versus homologous temperature for various processing times.](image)

**Figure 4.** Relative density versus homologous temperature for various processing times\textsuperscript{53} (in hours).

An important feature of the fiber fracture model is its dependence on densification; since the current density determines the number of bend cells, the distribution of cell lengths and the deformed heights of all unit cells, the density is used as input to the fiber fracture model (instead of the applied pressure and temperature). This coupling of models is only one-way of satisfying this need; experimental densification data could also be used. The densification response is assumed not to depend on the number of fibers fractured.
Figure 5. A representative unit cell used to calculate the probability of fiber fracture during consolidation processing. Note how increasing the inelastic compliance of the matrix or the fiber's rigidity reduces the likelihood of fiber fracture\(^{(57)}\).

There are many ways to present the outputs of a fracture model. One is to compute the number of breaks occurring per meter of fiber for a process conducted at a fixed densification rate (requiring continual change of applied pressure) and temperature. Figure 6 shows an example of this for the Ti-24Al-11Nb/SCS-6 system. Note how reducing the rate of densification (slow pressurization rate) and increasing the temperature reduces the damage.

### 4.3 Reaction Zone Growth

At elevated temperatures, titanium alloy matrices always react with reinforcing fibers and their protective coatings, eventually degrading the strength of the fiber and adversely affecting its interfacial properties. For example, it has been shown that in the Ti-24Al-11Nb/SCS-6 system, the sliding resistance of the fiber/matrix interface becomes too great (so that damage tolerance is lost) once the thickness of the reaction zone exceeds a critical value of about 1.5\(\mu\)m\(^{(58)}\). Based on studies of several titanium matrix composite systems, including Ti-24Al-11Nb/SCS-6 and Ti-6Al-4V/SCS-6, the thickness of the reaction zone has been shown to follow a simple parabolic law with respect to time:

\[
\delta = k t^{1/2}
\]

where \(k\) obeys an Arrhenius relationship of the form

\[
k = k_0 \exp \left( -\frac{Q}{RT} \right)
\]

(7)  

(8)
in which \( k_0 \) is the pre-exponential reaction (diffusion) mobility, \( Q \) is the activation energy for the reaction and \( T \) is the absolute processing temperature.

![Diagram of contour map showing lines of constant damage in a densification rate - homologous temperature space](image)

**Figure 6.** A contour map showing lines of constant damage in a densification rate - homologous temperature space\(^{(57)}\).

The reaction zone growth rate is obtained by differentiating equation 7 and substituting for \( k \) using equation 8:

\[
\delta = \frac{k_0 \exp\left(\frac{Q}{RT}\right)}{2\delta}
\]

(9)

### 4.4 Process Simulation

Models such as those described above, which relate processing conditions to changes in microstructure, may be combined to simulate the evolution of the microstructural "state" of a material during processing\(^{(60)}\). The process simulation can be thought of as the virtual process; it uses the models for microstructural evolution (in this case relative density, fiber fracture and reaction zone growth), "admissible" input schedules (i.e. schedules that are constrained by the limitations of a hot isostatic press or vacuum hot press) and material parameters required by the process models, to predict the microstructure that results from any process cycle.

The process variables during HIP’ing or vacuum hot pressing, i.e. the inputs to the simulation, are the temperature and applied pressure. The inputs (\( T(t) \) and \( P(t) \)) are time-dependent functions whose absolute values and slew
rates are constrained to lie between upper and lower limits (determined by the particular machine used).

\[ T_{\min} \leq T \leq T_{\max} \]  
\[ \dot{P}_{\min} \leq \dot{P} \leq \dot{P}_{\max} \]  
\[ \dot{T}_{\min} \leq \dot{T} \leq \dot{T}_{\max} \]  
\[ \dot{\rho}_{\min} \leq \dot{\rho} \leq \dot{\rho}_{\max} \]  

The simulation provides as output, the microstructural state variables as a function of time including the composite’s relative density \((D)\), the cumulative number of fibers fractured \((N_f)\) and the reaction zone thickness \((\delta)\). Put another way, the simulation allows one to trace the path along which the microstructure evolves in a space whose axes correspond to the microstructural state variables.

### 4.5 Simulation Results

The simulation tool has been used to conduct numerical “experiments” as a way of identifying the relationships between the process conditions and the microstructural state of the composite\(^{(60)}\). To illustrate the approach, results are presented for the consolidation of a conventional titanium alloy (Ti-6Al-4V) composite and one based on the \(\alpha_2\) intermetallic system (Ti-24Al-11Nb) both reinforced with SCS-6 fibers.

Figures 7-9 show the simulation results for a series of process cycles in which either the consolidation temperature or consolidation time have been systematically varied. Consider the \(\alpha_2\) matrix system of Figure 7 first. It shows that as the consolidation temperature is increased from 1100 to 1300K (while the slew rate for temperature is kept constant at 1°(K/sec) for each simulation), the composite’s relative density at the end of the cycle also increases (ideally, it should be close to unity). Raising the temperature also results in a decrease in the number of fractured fibers but an increase in the thickness of the fiber-matrix reaction product. A small amount of damage may be tolerable for some applications, but reactions that result in more than about a 1μm thick reaction product result in unacceptable frictional sliding behavior\(^{(58)}\). Figure 8 shows the effect of changing the “soak” pressure for a 1300K temperature, and again we see an increase in density as the pressure is increased, but too much interfacial reaction would occur before full density is achieved. Thus, none of the cycles shown in Figures 7 or 8 would result in a satisfactory composite.
Figure 7. Simulated consolidation of Ti-24Al-11Nb/SCS-6 system at 100MPa showing effect of changing the “soak” temperature\(^{(60)}\).

Figure 8. Simulated consolidation of Ti-24Al-11Nb/SCS-6 system at 1300K showing effect of changing the “soak” pressure\(^{(60)}\).

This can be contrasted to the Ti-6Al-4V matrix system in Figures 9 & 10. Here we again see similar trends with the process variables, but in this system both minimal fiber fracture and acceptable levels of chemical reaction can be accomplished with a 1100K/100MPa cycle. Lowering the pressure further reduces both the number of fiber breaks but also lowers the final density.
These simulation tools allow one to quickly explore “what if” questions and to use trial and error methods to rapidly converge on acceptable process cycles for the systems that can be processed successfully. They also allow the early determination of those that cannot! The tool also allows one to systematically explore the influence of the material system’s initial state on processing (i.e. on the final state of the previous process step). For example, increasing the fiber’s reference strength or decreasing the roughness of the monolayers widens the window of processibility and may enable the design of successful process cycles even for the $\alpha_2+\beta$ matrix composite system\cite{57}. 

Figure 9. Simulated consolidation of Ti-6Al-4V/SCS-6 system at 100MPa showing the effect of changing the “soak” temperature\cite{60}.

Figure 10. Simulated consolidation of Ti-6Al-4V/SCS-6 system at 1000 K showing the effect of changing the “soak” pressure\cite{60}. 

5. PROCESS PATH PLANNING

The use of simulation tools enables one to avoid costly and time consuming experimental trial and error during process design, but to this point it is still trial and error, and no guarantee can be made that the human intuition will lead to the optimum process. One ought to be able to do better using formal optimization methods and find truly the best process cycle for a given set of initial conditions, material properties, machine dynamics and desired product goal state. Many different approaches have been developed by the process control community for path optimization. Vancheeswarran et al. have explored one promising approach known as Generalized Predictive Control (GPC) which has the advantage of enabling one to fully exploit the predictive capabilities of process models.

GPC algorithms seek the inputs to a plant (the material and machine dynamics referred to in Figure 1) that drive the system’s state variables (i.e. the microstructure) to a desired goal state. This approach was chosen for study because the material’s response to process stimuli vary greatly from between the beginning and end of a process cycle. Many other (considerably simpler) approaches could be used instead of GPC if the materials dynamics did not change during the process. The GPC method is a self-tuning technique encompassing a multi-step predictor to achieve a more robust control. It uses a receding horizon philosophy in which the controller predicts and accounts for the changes in the controlled variable (density, number of fiber fractures, reaction zone thickness,...) that will occur in the future using present process knowledge and candidate controller actions.

The optimization problem that needs to be solved is schematically shown in Figure 11. At any moment in the consolidation process, the material’s state can be represented by a point in a relative density, reaction zone thickness, fiber deflection (i.e. probability of fiber fracture) state space. It will have reached this point along some path originating from the lower left in the figure. The consolidation process is irreversible, and so all future paths that are reachable by changing the machine conditions are enclosed in a generalized cone. The best (optimum) process path in state space will take the current material to the goal state.

To implement the GPC approach, one must know the dynamic responses of a) the machine used for consolidation and b) the material being consolidated, Figure 12. The predictive models discussed above serve the latter purpose well. The dynamic response of the HIP machine can sometimes exhibit complexity (e.g. coupling between temperature and pressure) but for exploring the process path optimization approach, this can be considerably
simplified. To illustrate the approach, Vancheeswaren et al\(^{(60)}\) have assumed that the machine dynamics, \(x_m\), are ideal:

\[
x_m = \eta = \begin{bmatrix} T \\ p \end{bmatrix}
\]

(14)

\[
\frac{d\eta}{dt} = \dot{u} = \begin{bmatrix} \dot{T} \\ \dot{p} \end{bmatrix}
\]

(15)

Therefore, the controller output is assumed to be the commanded values and rates, subject to satisfying the processing equipment's (limitations) constraints. The equipment constraints can be thought of as placing upper and lower limits on the pressure and temperature and their slew rates:

\[
\dot{T}_{mc} \leq \dot{T} \leq \dot{T}_{mh}
\]

(16)

\[
\dot{P}_{md} \leq \dot{P} \leq \dot{P}_{mp}
\]

(17)

\[
P_{min} \leq P \leq P_{max}
\]

(18)

\[
T_{min} \leq T \leq T_{max}
\]

(19)

where \(\dot{T}_{mh}\) and \(\dot{T}_{mc}\) are the maximum heating and cooling rates, respectively, and \(\dot{P}_{mp}\) and \(\dot{P}_{md}\) are the maximum pressurizing and depressurizing rates respectively.

The consolidation model can be written:

\[
\dot{x}_h(t) = F(x_h(t), \eta(t))
\]

(20)

\[
0 \leq G = (x_h(t), \eta(t))
\]

(21)

where

\[
\dot{x}_h(t) = F(x_h(t), \eta(t))
\]

(22)

\[
x_h(t) = \begin{bmatrix} D(t) \\ v_j(t) \\ r(t) \end{bmatrix}
\]

(23)

represents the materials dynamic response and

\[
\eta(t) = \begin{bmatrix} T(t) \\ P(t) \end{bmatrix}
\]

(24)
Figure 11. The process path optimization problem encountered in consolidation processing of metal and intermetallic matrix composites\(^{(61)}\).

Here \(x_h(t)\) is a state vector whose components are the relative density of the material's s-lamina \(D(t)\), the deflection of a fiber in the i-th unit cell \(v_i(t)\), and the fiber's reaction zone thickness \(r(t)\). \(\eta(t)\) is defined as the environment vector, whose components are the applied temperature and pressure. The vector field \(F(x, \eta)\) is complicated and is given by the time dependant mechanisms of the states described earlier. It can be written:

\[
F(x_h, \eta) = \left[ \sum_{i=1}^{\delta} (\xi_i P L C_i(x_h, \eta) + D i f_i(x_h, \eta)) \right] \quad \text{(25)}
\]

\[
(g(D, \eta) - h(v_i, T))_i \\
r(r, T)
\]
CONTROL SYSTEM BLOCK DIAGRAM

\[ u = \begin{bmatrix} T_{\text{rate}} \\ P_{\text{rate}} \end{bmatrix} \]

\[ \eta = \begin{bmatrix} T \\ P \end{bmatrix} \]

\[ x_n = \begin{bmatrix} D_n \\ V_n' \\ r_n \end{bmatrix} \]

Figure 12. The architecture of the composite consolidation control problem. The vectors \( u \) and \( \eta \) represent the plant controls. \( D_n, V_n' \) and \( r_n \) are the materials (time evolving) density, the fiber deflection suffered by the \( i \)-th unit cell and the thickness of fiber-matrix interface\(^{60}\).

Here the time dependant densification mechanisms due to power law creep are denoted \( PLC_i(x_n, \eta) \) and for diffusional flow by \( Dtf_j(x_n, \eta) \). The densification of the composite is modeled as two distinct layers, the \( s \) and the \( r \) layers, which are considered as independent states in the non-linear simulation. Since the density of the \( s \)-layer is modeled with two different geometries (Stage I and II), the smoothing functions described in Ref. \((53)\) are also incorporated. The inequality constraint \( G(x, \eta) \) is used as a way to introduce the plastic yielding (instantaneous) contribution to densification. Plasticity is modeled as a time independent mechanism (that is the densification response to an applied stress occurs instantaneously) and so the resulting vector field has finite discontinuities, which considerably compounds the problem.

5.1 Optimization Methodology

Implementation of the optimization scheme has been achieved by calculating the states (using locally linearized models) at discrete (2 minute) intervals during the process, out to a “look-ahead” horizon of 40 minutes (20 sampling intervals, \( N_t \)). An optimal process variable schedule \( \eta(t) \) can then be designed by perturbing the actuator input slew rates (which are constrained by the machine) such that they simultaneously lie in their admissible space and minimize an objective function which is a weighted function of the squared (Euclidean) distance between the projected future material states (based on the
linearized model) and a user defined goal state. The function, $E$, to be
minimized is:

$$E = \frac{1}{N_t} \sum_{k=1}^{N_t} k^2 [x(k) - x_g]^2$$

(26)

The objective function is quadratic and uses an aggressive strategy by
weighting the future more heavily than the present (because of $k^2$ in Eq. 26).
The specifications (constraints) and the objective function are easily proven to
be convex and therefore the (locally) optimal temperature and pressure slew
rates can be found by convex programming. The constraints in the convex
optimization problem can be set up as a set of Kuhn-Tucker Equations and
solved using the MATLAB optimization toolbox(62).

A sequential quadratic programming (SQP) method is then used to
solve the problem and is guaranteed superlinear convergence by using a quasi-
Newton updating procedure for accumulating second order information. Once
the optimal values of the temperature and pressure slew rates are found, they
are used as inputs to the simulation (non-linear models) which is then
integrated forward by one sampling interval (2 minutes). A linearization is then
conducted about this new operating point and the process is repeated.

Figures 13 & 14 show examples of optimal temperature and pressure
cycles calculated using the GPC method together with the predicted temporal
evolution of the state vector components. The goal state for this system was a
relative density of one, a reaction product thickness of 0.6μm and zero
deflection for each of the unit cells. The HIP machine was assumed to have a
maximum operational temperature and pressure 1100K and 100MPa, a
temperature slew rate of 1K/s and pressurization rate of 0.022MPa/s. This
“good” result was obtained by also applying a time delay constraint on the
pressure slew rate. When this is not included, the GPC designed cycle closely
approaches the goal state density and reaction layer thickness but incurs
significant fiber fracture very early in the process. This happens because the
controller fails to increase the machine’s temperature fast enough to soften the
matrix and avoid large fiber deflections. Effects like this can also be overcome
by increasing the weighting attached to fiber damage contributions to the
current state - goal state vector (Eq. 26) during the convex optimization step.
6. SENSOR TECHNOLOGY

The direct measurement of a component's density throughout a consolidation process cycle allows one to rapidly evaluate the validity of consolidation models\(^{(63)}\). It also enables one to implement feedback control about a planned path in state space provided the models for fiber fracture and reaction zone growth are reliable. The reliability of the fracture model depends on the reliability of densification kinetics and the assumed strength properties for the fiber. Since the former could be a directly sensed quantity, the feedback control approach requires only reliable fiber strength data and kinetic parameters for fiber-matrix reactivity. Both can be obtained to more than adequate precision with a few simple experiments.

During consolidation processing no mass change occurs and so the relative density can be obtained by making measurements of the volume or shape of the component during consolidation. This can be done using multifrequency eddy current techniques\(^{(21, 22, 63)}\). Probe-type sensors like those shown in Figure 15 have been used for measuring the separation distance between a fixed probe-tip and the (changing) position of the sample surface near the probe tip\(^{(22)}\). Pairs of probes maintained a fixed (or known) distance apart then allow determination of a component's thickness. In a sense, they become eddy current calipers. Other probe array configurations can give the dimensional changes of even complex shaped parts if this is necessary.

The response of these eddy current probes depends upon the probe's design/operating frequency, the electrical/magnetic properties of the component, and the component's shape in the vicinity of the probe. Relationships between these parameters and the impedance of the probe/test circuit have been investigated to perfect the sensor's design and develop algorithms to analyze multifrequency impedance data so that the component's dimensions may be deduced to the needed level of precision for model verification or feedback control. For axisymmetric configurations, the analytical approach of Dodd and Deeds\(^{(64)}\), together with a network analysis of the test circuit, may be used to derive the fundamental relationships required. For samples of more complex shape, an electromagnetic finite element method can be used\(^{(65)}\).
Figure 13. Example of a calculated optimal temperature/pressure path and resulting microstructural evolution for the Ti-24Al-11Nb/SCS-6 system\textsuperscript{(61)}.

Figure 14. Example of a calculated optimal temperature/pressure path and resulting microstructural evolution for the Ti-6Al-4V/SCS-6 system\textsuperscript{(61)}.

Normally, a two-coil design for each probe (as schematically indicated in Figure 13) is used so that the measured response is, to first order, independent of temperature (this avoids the need for active cooling of the sensor which is impractical in the HIP environment). The primary coil of the sensor, Figure 14, is connected to a variable frequency oscillator with optional power amplification. The current ($I_p$), that flows in the solenoid generates an electromagnetic field, a fraction of which links the sample of interest and the secondary coil. By measuring the voltage drop ($V_p$), across a precision resistor ($R$) to ground, the value of the primary current ($I_p$) can be continuously monitored. The fluctuating electromagnetic field associated with the primary
current induces eddy currents in the nearby conducting sample. The eddy currents, whose magnitude depends upon the rate of change of flux (which increases with frequency), act to oppose the change in field (Lenz's law) and thereby perturb the field of the primary coil. The perturbed field can be conveniently measured with a second coil aligned coaxially with the primary. The voltage induced in the secondary coil ($V_s$), though small can be amplified, and if measured with a high impedance instrument, is independent of the secondary windings resistance, and thus the sensor's temperature.

\[ V_s = M \frac{dN_p}{dt} \]

**Figure 15.** Schematic illustrations of the use of eddy current probe sensors to measure shape (volume) changes during consolidation.

The ratio $V_s : V_p$ defines a sensor gain ($G$), and the difference in phase between primary and secondary, $\phi = \phi_s - \phi_p$, defines the measured phase. Then at any measurement frequency, the complex impedance components can be found from:

\[
\text{Real}Z = \left( \frac{G}{G_0} \right) \sin(\phi - \phi_0)
\]

\[
\text{Imag}Z = \left( \frac{G}{G_0} \right) \cos(\phi - \phi_0)
\]

where the subscript zero refers to the values of an empty coil located far from any conductor.
The impedance normalized by the empty coil value is plotted on the right in Figure 16 for two different distances h above a uniform conductor with the electrical conductivity of copper. At low frequencies, the rate of change of flux is small, and the eddy current density is low, but is distributed deeply within the sample because the skin depth δ is large (recall that δ = (2/ωσμ)½ where ω is the radial frequency, σ the conductivity and μ the sample's permeability). At low frequencies there is quite a large change of phase because the flux deeply penetrates the conductor, but almost no eddy current losses occur because the eddy current density is small, so the impedance is almost purely inductive (imaginary). As the frequency is increased, eddy current densities increase resulting in greater losses that are reflected by the increase in the real part of Z. The imaginary component decreases because the flux does not penetrate as deep into the sample. At very high frequencies, the skin effect limits the depth of flux penetration severely, and the total eddy current losses also decrease because the volume supporting the eddy currents becomes very small, and overpowers the increase in current density associated with the now very high rate of flux change.

**EDDY CURRENT SENSOR PRINCIPLE**

![Diagram of eddy current sensor principle](Image)

**Figure 16.** The eddy current probe sensors measurement principle.
In the limit, the eddy current losses go to zero and the impedance curve intersects the imaginary axis at a value determined by the ratio of the flux linking the sample and the secondary coil to the total flux. Thus, as the sample densifies, and its surface moves away from the sensor (see for example the curve for h=4.8mm), fewer flux lines link the sample and the high frequency intercept moves towards the origin at \( Z=0+1j \).

Using an axisymmetric finite element model the sensor configuration shown in Figure 15 has been analyzed in detail\(^{22}\). The analysis consists of solving for the unknown magnetic vector potential \( \Delta \) over a range of frequencies for different lift-offs and sample conductivities and then computing the coil impedances. Model parameters such as coil diameter, coil length, the number of turns, the number of secondary coils and their placement, sample thickness, material parameters and loading conditions can then be easily studied, and optimized sensors designed for various applications. This has all been significantly simplified by the advent of user friendly electromagnetic finite element codes\(^{65}\).

The governing equations for these types of problems follow from Ampere’s Law:

\[
\nabla^2 \Delta = -\mu J
\]

(29)

where \( \mu \) is the magnetic permeability, \( \Delta \) the magnetic vector potential, and \( J \) is the total current density. It is composed of two contributions:

\[
J = J_{\text{eddy}} + J_{\text{coil}}
\]

(30)

The induced eddy current density within the sample can be written as,

\[
J_{\text{eddy}} = \sigma E = -\frac{\sigma \partial \Delta}{\partial t}
\]

(31)

where \( E \) is the electric field and \( \sigma \) is the electrical conductivity. Substituting for \( J \) in Amperes Law gives the governing differential equation:

\[
\nabla^2 - \mu \sigma \frac{\partial \Delta}{\partial t} = -\mu J_{\text{coil}}
\]

(32)

With sinusoidal excitation,

\[
J_{\text{coil}} = J_{\text{coil}} e^{-j\omega t}
\]

(33)

and,

\[
 \Lambda = \Lambda e^{-j\omega t}
\]

(34)

Thus, the governing equation reduces to the fundamental eddy current relation,
\[ \nabla^2 A + j \omega \mu \sigma A = \mu I_{\text{coil}} \]  

(35)

The boundary conditions are that \( A \) and its normal derivative are continuous across each boundary.

The impedance measured experimentally depends upon the average value of the vector potential at the location of the secondary coil:

\[ Z = \frac{4\pi^2 N_s r_f}{l_p} [\text{Im}(A_{\text{ave}}) - j \text{Re}(A_{\text{ave}})] \]  

(36)

where \( f \) is the excitation frequency (Hz), \( N_s \) is the number of secondary coil turns, \( r_s \) is the secondary coil radius and \( l_p \) the primary coil current.

One usually measures an impedance normalized by the impedance of the probe when it is located far from a sample. This empty coil impedance, \( Z_0 \), is given by:

\[ Z = \frac{4\pi^2 n_s r_f}{l_p} [\text{Im}(A_0) - j \text{Re}(A_0)] = R_0 + j\omega L_0 \]  

(37)

where \( A_0 \) is the vector potential in the absence of the sample, \( R_0 \) is the equivalent resistive impedance component and \( \omega L_0 \) the empty coil’s inductive reactance component. In general \( \omega L_0 \gg R_0 \) and so the normalized impedance,

\[ Z_n = \frac{Z}{R_0 + j\omega L_0} = \frac{R}{\omega L_0} + \frac{j\omega L}{\omega L_0} = \frac{[-\text{Im}(A_{\text{ave}}) + j \text{Re}(A_{\text{ave}})]/\text{Re}(A_0)}{R_0 + j\omega L_0} \]  

(38)

For a differential sensor (i.e. one with two spatially separated opposingly wound pick-up coils), the impedance is calculated by algebraically summing the impedance of the individual secondaries.

The normalized high frequency impedance for a probe coil with a single secondary is shown versus lift-off in Figure 17 (curve 4). One sees that for larger separation distances, the effect of a small further change in separation distance becomes progressively more difficult to observe. In practice, one is interested in the final stages of shrinkage of a sample when the surface has moved \(~10\%\) of the sample thickness away from the probe. If the probe diameter is substantially less than the sample thickness results in a loss of sensitivity to the elimination of a sample’s final porosity.

One way to overcome this limitation is to increase the sensitivity at larger separation distances by increasing the sensor’s diameter. But this increases the sensitivity to concurrent changes in lateral sample dimensions
(length and width). An alternative approach involves the use of a differential secondary configuration. Figure 17 shows the calculated normalized impedance for several differential sensor designs (shown on the right) with secondary coil separations of 3.2, 6.4, and 9.6mm. The normalization practice now inverts the impedance plane diagram but one notes that there is now a much stronger effect of impedance upon lift-off.

The size of the impedance curve, can be controlled by varying the secondary coil separation. The effect of this separation upon the normalized imaginary Z-component verses lift-off relationship is shown in Figure 17. We see that the sensitivity to lift-off (i.e., the slope of the curve) is determined by the number and separation of the secondary coils. In particular, we note that there is considerable enhancement in far field sensitivity (i.e., lift-offs greater than 4mm) for these differential sensor geometries.

Figure 17. Calculated sensor responses for different eddy current sensor probe designs$^{22}$.

Based upon these results, sensors have been designed using boron nitride preforms and high temperature (e.g., platinum or molybdenum) windings and used to measure changes in the thickness of composite samples to better than 20μm precision during HIP cycles to temperatures in excess of
1000°C. They have been used both to evaluate the predictions of densification models and for on-line process control. The accuracy appears to be limited by thermal effects upon the dimensions of the sensors and changes to the electrical conductivity of its components and the sample as the temperature varies. Further work is needed to understand the subtle consequences of these.

7.0 FEEDBACK CONTROL

The feedback control of microstructural states during composite consolidation is a formidable problem. The optimal controller will vary throughout the process, the dynamics are highly nonlinear, irreversible, and only some of the states are sensed. However, because the dynamics of composite consolidation are fairly slow (the process requires several hours to complete), a relatively small closed-loop bandwidth is required for successful control. Hence, the Nyquist sampling rate for the sensors is low and there is ample time (1-2 minutes) for computations in a control loop. In view of this, and given the difficulty of the feedback control problem, it has been logical to consider controller architectures that might normally be too computationally intensive for processes requiring faster sensor sampling rates.

One that may be suitable for this type of application is shown in Figure 18. It was originally designed to allow the control of density (D) and grain size (G) during the consolidation of powders using models developed by Ashby and others(66). In view of the similarities between this and composite consolidation, it appears a good place to begin an attack of the composite problem. The architecture functions by constantly forming a local linearization of the material dynamics which is then used in a controller design procedure (in this sense it is similar to GPC path planning). The controller then attempts to establish a temperature and pressure schedule that steers the current material state, \( x_h \) to the desired goal state \( x_g \).

The architecture shown in Figure 18, uses what is known as an observer based controller. In this approach the observer (estimator) uses the linearized process models to estimate future material states (by applying Kalman filtering) given the present estimated states \( \hat{D}, \hat{G}, \hat{T}, \hat{P} \), the sensed states \( D,G,T,P \), the current actuator inputs \( \hat{T}, \hat{P} \), and a gain matrix \( L \). The observer based controller is then designed using linear quadratic regulator (LQR) theory. Here, the states (either estimated or sensed) are optimally fed back to the linearized system as inputs after they are acted upon by some gain matrix \( K \) to drive the system to the future material states. These problems have been rigorously studied and have well known solutions (to find \( K \) and \( L \)) involving
algebraic Ricatti equations. To ensure stable observation and control of the system, coprime factorizations of the plant and the nominal controller are computed using the matrices K and L with formulas found in refs. (68, 69). At this point, a stable nominal controller has been found (for some point in the process) which adjusts the actuator inputs (U) to reduce the effects of sensor and actuator noise, Figure 19. The observer based controller is then continuously redesigned using new local linearizations of the system, as the process progresses through the consolidation cycle.

To drive the regulated variables (D,G) to their goal (xₜ), a finite response Q filter is added to the system, Figure 19. W represents the set of exogenous inputs (the goal state (xₜ), the sensor noises and actuator disturbances), Z comprises the regulated variables (i.e. the controls, \( T, \dot{P} \), the machine states \( T,P \)) and the material states (D,G), U comprises the actuator inputs (\( \dot{T}, \dot{P} \)), and Y the sensed outputs (D,G,T,P and \( xₜ \)). The nominal controller is then modified or augmented to produce a signal \( e \) (with the same number of dimensions as the sensed outputs \( Y \)), and an auxiliary input signal \( v \) (of the same size as the actuator inputs U). The important point to this controller architecture is that the map from \( e \) to \( v \), \( H_e \), is forced to zero, so that when a finite impulse response filter \( Q \) is inserted, the overall transfer matrix \( G_{overall} \) assumes the form:

\[
G_{overall} = H_{zw} + H_{zw}QH_{ew}
\]

(39)

which will then have the affine structure in \( Q \) needed for convex optimization. This convex optimization results in the elements of the \( Q \) matrix \( Q_{ij} \), such that the sensor and actuator noises are minimized, the closed loop system is stable, and the regulated variables are driven to their goal state.

Figure 18. Feedback control architecture designed to control density (D) and grain size (G) during the HIPing of powders (66).
The convex program is set up to minimize some objective function (in this case a weighted sum of the Euclidean distance between the goal state and the projected future states) to select the “best” controller (locally). Moderately sized convex programs can be solved easily in the available time. Once Q is found, controller specifications are complete, and the control is calculated and implemented until the next sensor sampling of the process, whereupon the process is repeated. Figure 18 shows an example of a simulation conducted with this controller (assuming perfect sensing of D and G). The goal states are shown in dashed lines, and it is clear that for this case at least, the controller is able to practise the principles of IPM control.

Figure 19. System after closing loop with nominal controller.

This approach to feedback control has now begun to be applied to HIP consolidation processing of composites with encouraging initial results\(^{(67)}\). The HIP consolidation of metal alloy powders/composites to a goal state microstructure occurs by similar mechanisms to other deformation processes such as vacuum hot pressing, isothermal forging, hot rolling, extrusion, superplastic forming/diffusion bonding,... These feedback control concepts may be a logical starting point for designing controllers for these processes also.
8.0 SUMMARY

Intelligent Processing of Materials is a new way of designing and controlling advanced materials synthesis and processing. It is making a pervasive impact across a broad spectrum of materials and processes. Predictive process modeling, combined with convex optimization techniques, are beginning to allow materials engineers to rapidly design processes that result in the attainment of a goal state microstructure — a key step in the development of a processing technology that will finally enable the high yield manufacture of truly engineered materials. When microstructure attribute sensors are available, a new (for materials synthesis/processing) type of IPM feedback control methodology becomes feasible. The first tentative steps in this direction have been made and at least one approach appears to hold promise. However, its application is paced by the emergence of new microstructure sensors. As the materials community finds ways to field noninvasive techniques based on eddy currents, laser ultrasonics, microwave reflectivity, ellipsometry, etc. to satisfy these sensor needs, the extension of IPM as a feedback control method is likely to rapidly grow in order to reduce process costs and realize the potentially significant improvements in the yield/quality of high performance materials.
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